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“Knowledge is such a treasure which cannot be stolen”

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IS : 64 -1972
(Superseding IS : 65 - 1950)
(Reaffirmed 2009)

Indian Standard
SPECIFICATION FOR
BARIUM SULPHATE PIGMENTS FOR PAINTS
(*First Revision*)

Third Reprint NOVEMBER 1998

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

AMENDMENT NO. 2 NOVEMBER 1987

TO

**IS : 64-1972 SPECIFICATION FOR BARIUM SULPHATE
PIGMENTS FOR PAINTS**

(First Revision)

[Page 5, Table 1, Sl No. (iv), col 2]:

a) Add the symbol '++' after oil absorption.

b) Add the following foot-note at the end of the table:

'++This shall, however, be within + 10 percent of the approved sample, if any.'

AMENDMENT NO. 1 FEBRUARY 1977
TO
IS : 64 - 1972 SPECIFICATION FOR BARIUM
SULPHATE PIGMENTS FOR PAINTS

(First Revision)

Alterations

[Page 5, Table 1, against Sl No. (ix)] -

a) Col 3 and 4 - Substitute 'Min, 4.4'
for '4.45'.

b) Col 5 - Substitute 'Min 4.3' for
'3.36'.

(Page 8, clause A-2, heading) - Substitute
'DETERMINATION OF ACID SOLUBLE SALTS EXPRESSED
AS BARIUM CARBONATE' for 'DETERMINATION OF BARIUM
CARBONATE'.

(CDC 50)

Indian Standard
SPECIFICATION FOR
BARIUM SULPHATE PIGMENTS FOR PAINTS
(*First Revision*)

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(Continued on page 2)

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Indian Standard

SPECIFICATION FOR BARIUM SULPHATE PIGMENTS FOR PAINTS

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 18 May 1972, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

0.2 The revision has been brought up taking cognizance of the technological advancements registered in the manufacture of pigments and covers both barytes and blanc fixe. In the revision new requirements like particle shape, pH of aqueous extract and relative density have been prescribed. This standard supersedes IS : 65-1950*.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-19601. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for barytes (naturally occurring barium sulphate) and blanc fixe (the precipitated barium sulphate). The material is intended for use as extenders for paints.

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions given in 2 of IS : 33-1963‡ and IS : 1303-1963§ shall apply.

*Specification for blanc fixe for paints.

†Rules for rounding off numerical values (*revised*).

‡Methods of test for dry pigments and extenders for paints (*revised*).

§Glossary of terms relating to paints (*revised*).

3. TYPES

3.1 The material shall be of the following two types.

3.1.1 Type 1 material shall consist of naturally occurring barium sulphate which may be bleached and shall have two grades:

- a) Grade 1, and
- b) Grade 2.

3.1.2 Type 2 material shall be the precipitated barium sulphate.

4. REQUIREMENTS

4.1 Form and Condition — The material shall be in the form of dry powder or in such a condition that it can be reduced to the powder form by crushing, without grinding action, under a palette knife.

4.2 The material shall also comply with the requirements given in Table 1.

5. PACKING AND MARKING

5.1 Packing — The material shall be suitably packed as agreed to between the purchaser and the supplier.

5.2 Marking — The containers shall be marked with the following:

- a) Name of the material;
- b) Manufacturer's name or trade-mark, if any;
- c) Net mass of the material; and
- d) Month and year of manufacture.

5.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6. SAMPLING

6.1 Representative samples of the materials shall be drawn as prescribed under 3 of IS : 33-1963*.

*Methods of test for dry pigments and extenders for paints (*revised*).

TABLE 1 REQUIREMENTS FOR BARIUM SULPHATE PIGMENTS

(Clause 4.2)

SL No.	CHARACTERISTIC	REQUIREMENT			METHOD OF TEST, REF TO	
		Type 1		Type 2	Appendix	CI No. in IS : 33 1963*
		Grade 1	Grade 2			
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Compositions					
	a) Barium (as BaSO ₄), percent by mass, <i>Min</i>	95	95	97	A-1	—
	b) Carbonates (as BaCO ₃), percent by mass, <i>Max</i>	2.24	2.24	0.45	A-2	—
ii)	Volatile matter, percent by mass, <i>Max</i>	0.5	0.5	0.5	— 6	
iii)	Residue on sieve, percent by mass, <i>Max</i>	0.25 on 40-micron IS Sieve (400 mesh)	0.25 on 63-micron IS Sieve (240 mesh)	0.1 on 40-micron IS Sieve (400 mesh)	—	7
iv)	Oil absorption	← 6 to 12 →			15 to 30	8
v)	Particle shape	Characteristics of the material and similar to that of approved sample			B	—
vi)	Colour	A close match to that of approved sample			—	9
vii)	Matter soluble in water, percent by mass, <i>Max</i>	← 0.5 →			—	12
viii)	pH of the aqueous extract	← 6 to 8 →			—	14
ix)	Relative density† at 25/25°C	← 4.45 →			3.36	C —

*Methods of test for dry pigments and extenders for paints (revised).

†Synonymous with specific gravity.

7. TEST METHODS

7.1 Tests shall be conducted as prescribed in relevant clauses of IS : 33 1963* and in Appendices A to C. References to appendices and IS : 33-1963* are given in col 6 and 7 of Table 1 respectively.

*Methods of test for dry pigments and extenders for paints (revised).

7.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

8. CRITERIA FOR CONFORMITY

8.1 A lot shall be declared as conforming to the requirements of this standard if the test results of composite sample satisfy the requirements prescribed under 4.

APPENDIX A

[*Table 1, Item (i)*]

DETERMINATION OF BARIUM SULPHATE AND CARBONATES IN BARIUM SULPHATE PIGMENTS

A-1. DETERMINATION OF BARIUM SULPHATE

A-1.1 Outline of the Method — The material is fused with fusion mixture and the fused mass disintegrated with water. The barium carbonate formed is filtered off, washed free of any sulphate, dissolved in dilute hydrochloric acid and from this solution total barium, precipitated as sulphate, is weighed. Barium sulphate content is calculated after correcting the mass for the sulphate formed from the barium carbonate present in the pigment as determined in A-2.3.

A-1.2 Apparatus

A-1.2.1 Conical Flask — 250 ml.

A-1.2.2 Oven — Capable of maintaining temperature of $105 \pm 2^{\circ}\text{C}$.

A-1.2.3 Muffle Furnace — Capable of maintaining temperature up to 600°C .

A-1.2.4 Asbestos Pad Gooch Crucible

A-1.2.5 Desiccator

A-1.2.6 Platinum Crucible

A-1.3 Reagents

A-1.3.1 Hydrochloric Acid — Approximately 1 N (*see* IS : 265-1962†).

*Specification for water, distilled quality (*revised*).

†Specification for hydrochloric acid (*revised*).

A-1.3.2 Dilute Sulphuric Acid— Approximately 6 N (see IS : 266-1961*).

A-1.3.3 Anhydrous Sodium and Potassium Carbonates (Fusion Mixture) — 1: 1 by mass.

A-1.3.4 Dilute Hydrochloric Acid — 1: 1 by volume.

A-1.3.5 Ammonium Hydroxide Solution — of relative density 0.88.

A-1.3.6 Sodium Carbonate Solution — 3 percent (m/v).

A-1.3.7 Ammonium Sulphate Solution— 10 percent (m/v).

A-1.3.8 Methyl Red Indicator — 0.03 percent solution in water (m/v).

A-1.4 Procedure

A-1.4.1 Determination of Barium Sulphate — Weigh accurately about 0.5 g of the sample and mix with about 10 times its mass of fusion mixture using an agate mortar if necessary. Fuse the mixture in a platinum crucible for about an hour. Transfer the crucible, when partially cooled, to a 250-ml beaker containing about 100 ml hot water. Heat the beaker to disintegrate the melt and to get it completely dislodged from the crucible. Remove the crucible, after carefully washing it with a jet of water to free it from any adhering residue. Filter the contents of the beaker through a Whatman No. 2 or equivalent filter paper and wash the residue with the 3 percent hot sodium carbonate solution, till the washings give no positive test for sulphates. Transfer the residue to the (now empty) 250-ml beaker, by piercing the filter paper and washing the residue down with a jet of water. Wash down the filter paper with few ml of hot dilute hydrochloric acid and finally with hot water into the beaker. The residue will have been completely dissolved. Add few drops of methyl red indicator, neutralize excess acid by ammonia solution added dropwise, and make acidic again with few drops of dilute hydrochloric acid. Add water if necessary to make up the volume to about 150 ml and boil. Gradually add slight excess of the 10 percent ammonium sulphate solution and continue gentle boiling for 10 to 15 minutes more to render the precipitate granular and easily filterable. Stand the precipitate for some time and filter through a previously ignited and tared asbestos gooch crucible. Wash with hot water, heat in an oven for 15 to 20 minutes at $105 \pm 2^\circ\text{C}$, ignite at about 600°C in a muffle furnace for 30 to 40 minutes, cool and weigh.

A-1.5 Calculation

A-1.5.1 Barium sulphate, percent by mass = $\frac{100 M_1}{M_2} - 1.182 B$

*Specification for sulphuric acid (revised).

where

M_1 = mass in g of the residue in the gooch crucible,

M_2 = mass in g of the material taken for test, and

B = barium carbonate percent by mass as determined in A-2.3.

A-2. DETERMINATION OF BARIUM CARBONATE

A-2.1 Outline of the Method — Barium carbonate from a known quantity of material is extracted with dilute hydrochloric acid, precipitated is barium sulphate and barium carbonate calculated from the mass of the sulphate.

A-2.2 Procedure — Weigh accurately about 5 g of the material and transfer to a 250-ml conical flask. Add about 60 ml of hot dilute hydrochloric acid and carefully swirl the mixture clockwise and anticlockwise intermittently for 3 to 4 minutes. Allow to settle and carefully filter through a No. 2 Whatman or equivalent filter paper, maximum quantity of the supernatant solution into a 250-ml beaker. Give two more washes each of about 30 ml hot acid and a final wash of about 30 ml hot water, decanting these near the rim of the filter paper cone. Heat the solution in the beaker to boil and precipitate the barium as barium sulphate by gradual addition of hot dilute sulphuric acid. Continue gentle boiling for, 10 to 15 minutes to render the precipitate easily filterable. Stand for half an hour and filter through a previously ignited and weighed asbestos gooch crucible. Wash, dry, ignite, cool and weigh as in A-1.4.1.

A-2.3 Calculation — Barium carbonate, percent by mass = $\frac{84 \cdot 58 M_3}{M_4}$

where

M_3 = mass in g of the residue in the crucible, and

M_4 = mass in g of the material taken for test.

APPENDIX B

[Table 1, Item (v)]

DETERMINATION OF PARTICLE SHAPE

B-0. GENERAL

B-0.1 Outline of the Method — Prepare a thin film of the material on a glass plate and view through a microscope at 400 magnification.

B-1. APPARATUS

B-1.1 Microscope — a suitable one for viewing at minimum of 400 magnification.

B-2. PROCEDURE

B-2.1 Mix a small quantity, about 0.1 g of the material with petroleum hydrocarbon solvent and apply a thin film of the paste on a clean glass slide. The film should be free from any aggregate particle as far as possible. Observe the shape of the material under the microscope using 400 magnification. Compare the shape of the material with a similarly prepared slide of the approved sample. The shape of the material shall not be different from that of the approved sample.

APPENDIX C

[*Table 1, Item (ix)*]

DETERMINATION OF RELATIVE DENSITY**C-0. GENERAL**

C-0.1 Outline of the Methods — Two methods are prescribed for determination of relative density. Method 2 which gives accurate results should be used in case of dispute. Method 1 is used for routine testing.

C-1. APPARATUS AND REAGENTS

C-1.1 Pyknometer — of 50 ml capacity.

C-1.2 Water-Bath — to maintain $25.0 \pm 0.5^{\circ}\text{C}$.

C-1.3 Manometer — a suitable one.

C-1.4 Desiccator — a suitable one to withstand a vacuum of one atmosphere.

C-1.5 Vacuum Pump

C-1.6 Thermometer — a suitable thermometer having a range of 0°C to 60°C and an accuracy of 0.1°C (*see* IS : 4825-1968*).

C1.7 Weighing Bottle

C-1.8 Wetting Liquid — A liquid of low evaporation rate and narrow boiling range. Generally, white kerosine of low evaporation and boiling

*Specification for laboratory and reference thermometers.

range is suitable. Where kerosine is unsuitable, other wetting agents like glycerol, ethylene glycol, etc, may be used.

C-1.9 Bottle— A storage bottle (*h*, Fig. 1) for kerosine or other wetting liquid.

C-1.10 Bell Jar— A glass bell jar *b* with a two-hole rubber stopper. Into one hole of the stopper shall be fitted a separatory funnel with a well ground stop-cock (*c*, Fig. 1), extending into the pyknometer. Into the other hole of the stopper shall be fitted a glass tube with a well ground three-way stop-cock (*d*, Fig. 1) and connected with the vacuum pump (*e*, Fig. 1). The bell jar shall rest on a sheet of rubber, cemented or vulcanized to a glass or iron plate. With stop-cock *c* closed and stop-cock *d* open to the pump, the system shall maintain an absolute pressure of at most 3 mm Hg. A desiccator may be used instead of a bell jar.

C-2. PROCEDURE

C-2.1 Standardization of Pyknometer— Fill the pyknometer with freshly boiled distilled water and maintain at $25.0 \pm 0.5^\circ\text{C}$ and weigh after wiping the outside and making it dry. Empty the pyknometer, clean and dry and reweigh. Next fill the pyknometer with kerosine at $25.0 \pm 0.5^\circ\text{C}$, wipe, dry and weigh. Calculate the relative density of kerosine as follows:

$$\text{Relative density of kerosine } (P), \text{ at } 25/25^\circ\text{C} = \frac{M_1}{M_2}$$

where

M_1 = mass in g of kerosine, and

M_2 = mass in g of water.

C-2.2 Method 1 — Weigh about 10 g of the pigment after transferring to a clean and dry pyknometer. Add enough kerosine to the pyknometer to form clean layer approximately 6 mm above the pigment. Place the pyknometer in desiccator which shall be closed and attached to the water pump until the greater part of the air is removed from the system. Complete the operation in about 5 to 10 minutes. Close the system with a pinchcock and attach the desiccator to the oil pump for removal of small amounts of air given off at low pressures. Use the manometer to check the vacuum. When the absolute pressure is 3 mm Hg and constant, cut off the pump for short periods, taking care that the vacuum does not change materially due to leakage. Bubbles of air rise from the pigment very rapidly at first and decrease gradually and stop finally. When no more bubbles come, it may be assumed that occluded air has been removed and the material is wet with kerosine. Then slowly admit air to the desiccator by means of the pinchcock.

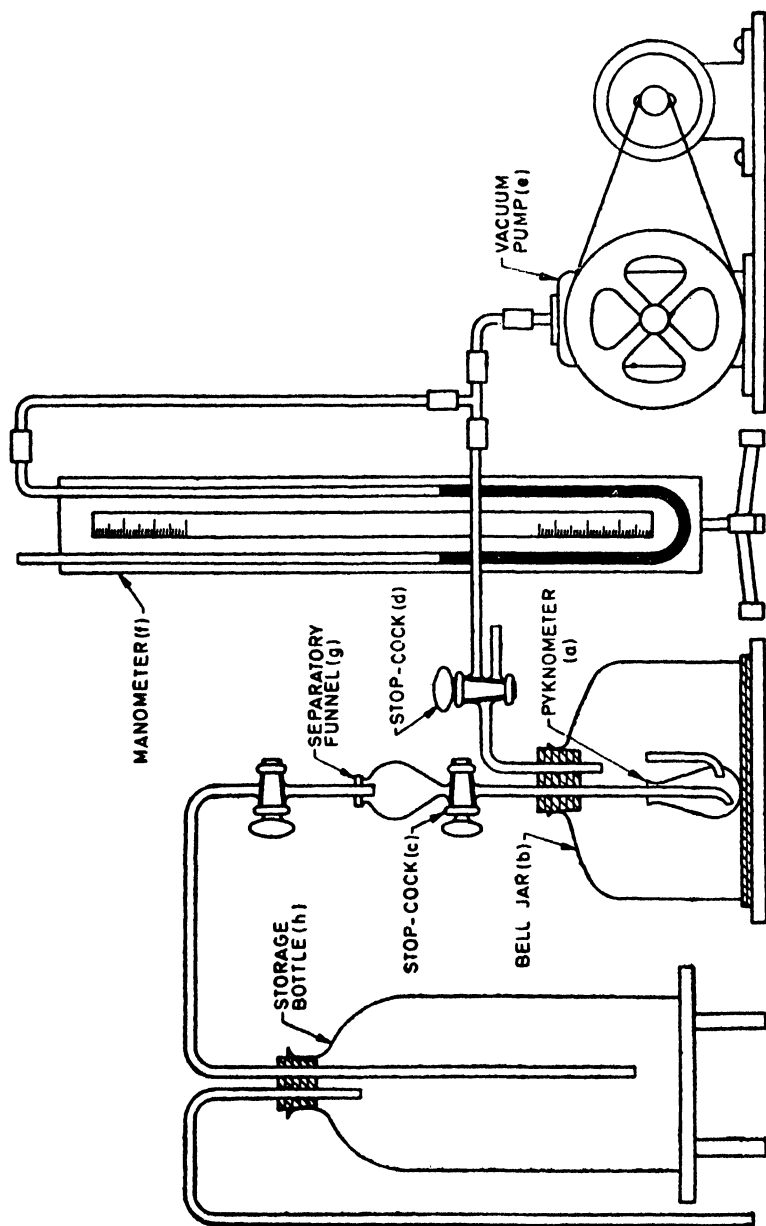


FIG. 1 APPARATUS FOR DETERMINATION OF RELATIVE DENSITY

C-2.2.1 Remove the pyknometer from the desiccator (bell jar), fill with kerosine at 24°C to 25°C, care being taken that sufficient quantity of kerosine is added to prevent air bubbles when the pyknometer is closed. Bring the pyknometer to $25.0 \pm 0.5^\circ\text{C}$. Carefully stopper the pyknometer, remove excess kerosine, wipe, dry and weigh.

C-2.3 Calculate the relative density as follows:

$$\text{Relative density of the material} = \frac{M_1 \times P}{(M_1 + M_2) - M_3}$$

where

M_1 = mass in g of material taken,

P = relative density of kerosine,

M_2 = mass of the bottle in g when filled with kerosine, and

M_3 = final mass of bottle in g when filled with kerosine and pigment.

C-2.4 Method 2 — The apparatus shall consist as shown in Fig. 1. Place the pyknometer containing the weighed, dried pigment under the bell jar. Close the stop-cocks *c* and *d* of the bell jar, start the vacuum pump, and then gradually open stop-cock *d* to the pump.

When an absolute pressure of 3 mm Hg has been attained and can be maintained, fill the separating funnel with kerosine, close stop-cock *d* and gradually open stop-cock *c*, adding sufficient kerosine to cover the pigment. Then stop the pump and release the suction at stop-cock *d*. Finally, fill pyknometer with kerosine, and complete the test as prescribed in C-2.2.1 and C-2.3.

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